Dye Removal Employing Coagulation and Flocculation

By

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Dissertation submitted in partial fulfillment of

the requirements for the

Bachelor of Engineering (Hons)

(Chemical Engineering)

JULY 2010

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CERTIFICATION OF APPROVAL

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Approved by,

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UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

July 2010

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

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NOR ADILLA BINTI RASHIDI

ABSTRACT

Currently, effluents which have been generated by the textile industry are one of the major issues that contribute to the water pollution problems. This is probably due to the fact that, this type of industry consumes high amount of process water and chemicals and therefore, produces high volume of highly polluted discharged water. Due to its dreadful impacts towards the environment and living organism, lots of researches have been carried out to treat these effluents. Presently, various methods have been established by the experts such as the biological, chemical and physical treatment. Associated with that, coagulation and flocculation which is one of the physical-chemical techniques are widely employed to treat those wastewaters. While implementing this method, the major concerns are on how to make the process efficient in terms of economic and residual water quality and at the same time, to improve the produced sludge (in terms of quantity and size characteristics) so that it will be easily treated or disposed later on. Besides, the coagulation and flocculation method was tested for Eriochrome Black T synthetic wastewater. The experiments had been carried out by varying few parameters, which were concentration of coagulant (aluminium sulphate), pH, temperature and effect of coagulation aids (bentonite and kaolin clay). Due to that, series of jar tests and shaking water bath had been carried out with 2 minutes mixing time at 200 rpm, followed by 30 minutes mixing time at 30 rpm and 1 hour of sedimentation process. Afterwards, the supernatant liquid was evaluated in terms of pH, Chemical Oxygen Demand (COD) and turbidity. Throughout the experiments, the optimum condition for the process was at 250 ppm of alum, pH 7 at operating temperature, 30^oC and bentonite concentration was at 750 ppm.

ACKNOWLEDGEMENT

Apart from the efforts of me, the success of any project depends largely on the encouragement and guidelines of many others. Thus, I would like to seize this opportunity to express my gratitude to the people who have been actively involved in the successful completion of this final year project.

First and foremost, I would like to express my deepest appreciation to supervisor, Dr Usama Mohamed Nour Eldemerdash for his most precious and valuable supervisions, advices and constructive criticisms from the very early stage of the final year project as well as giving me an extraordinary experience throughout the research period. Above all and the most needed, he provided me with unflinching encouragement and support in many ways.

Besides that, I would also like to offer my earnest appreciation to all the laboratory technicians for their full support during the research and experimental works. Without their guidance and persistent help, the research and experimental would not have been possible to accomplish effectively.

Last but not least, I am indebted and grateful to the family members and friends for the guidance and tremendous support received. Essentially, all of their crucial contributions and kindness are incredibly vital for the success of the project.

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CHAPTER 1

PROJECT BACKGROUNDS

1.1 Background of study

Textile industry is said to be one of the most complicated industries among all of the manufacturing industry and a considerable major source of the environmental contamination. This is due to its nature of textile wastewater, which consists of various waste chemical pollutants and simultaneously, consumes a large amount of fresh water[1]. In fact, in a real industry, it has been found that major of the effluents are generated from the material preparatory processing, dyeing and in finishing[2]. Thus, throughout the experimental works, dyestuff is selected as the pollutant. In addition, this dyestuff will contribute to high values of Chemical Oxygen Demand (COD), total dissolved solids and also turbidity in most cases. Thus, if these textile effluents are not well-treated, then it may cause various harm and damage to the river and also to the surrounding people. Besides, following figure illustrates the characteristics of the textile wastewater in the real application.

Table 1.1: Typical textile effluent characteristics[3]

Determinand	Woven fabric finishing	Knit fabric finishing	Stock and yarn dyeing and finishing
BOD[1] (mg/litre)	550 - 650	250 - 350	200 - 250
Suspended solids (mg/litre)	185 - 300	300	50 - 75
COD[2] (mg/litre)	850 - 1 200	850 - 1 000	524 - 800
Sulphide (mg/litre)	3	0.2	0 - 0.09
Colour (ADMI[3] units)	325	400	600
pH	7 - 11	6 - 9	7 - 12

[1] Biological oxygen demand [2] Chemical oxygen demand [3] American Dye Manufacturers Institute

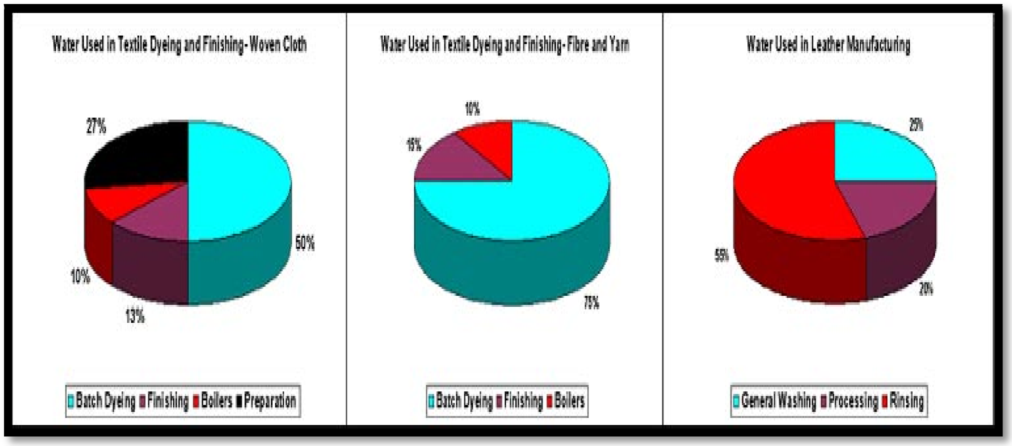

Figure 1.1: Water used in textile dyeing and finishing[4]

At present, there are numerous technologies which have been applied in treating the wastewater – biological, physical and chemical methods. However, for this research, coagulation and flocculation has been chosen as the main process in determining the effectiveness of dyes removal from the wastewater.

1.2 Problem statements

By applying the coagulations and flocculation methods in treating these textile effluents which mainly focus on the dyestuffs, several parameters' value are required to be investigated into detail. The parameters include temperature, pH, dosage of the coagulants and also the effect of coagulant aids. This is because; the coagulation and flocculation process will react efficiently at optimal conditions only. Even though coagulation and flocculation techniques are frequently employed in the industry, but little data is known about the optimal operation conditions of the process[5]. Plus, the optimal operation conditions for different types of dyestuff might be different from each other. In addition, optimization of these parameters will considerably improve and enhance the treatment process.

Apart from that, mechanical flocculation and chemical coagulation are among the primary wastewater treatments. Thus, optimal conditions need to be achieved so that it will simplify the rest of treatment process (secondary and tertiary wastewater treatments). Besides, our major concern through the implementation of this method is to produce an efficiency process (in terms of residual water quality after treatment and economic perspective) and to produce sludge which can be easily utilized or eliminated later on[6]. Principally, the main problems when dealing with dyes effluents is that, the dyestuffs must be removed completely since they can still be visible even at a very low concentration values[7].

1.3 Objective and scope of study

The scope of the research mainly concentrates on the coagulation and flocculation method in eliminating dyestuffs, which is one of the major contaminants consumed in the textile industry. In the meantime, the key objectives of conducting the research are like following:

i. To investigate the efficiency of coagulation and flocculation process as the first treatment in handling wastewater containing dyestuff.
ii. To conduct a jar test on the test water in order to estimate an optimum dosage of coagulant (aluminium sulfate) for the coagulation and flocculation process.
iii. To conduct a jar test on the test water in order to estimate an optimum pH for the coagulation and flocculation process.
iv. To conduct a jar test on the test water in order to estimate an optimum temperature for the coagulation and flocculation process.
v. To observe the effect of addition of coagulation aids (bentonite clay and kaolinite clay) during the coagulation and flocculation process.
vi. To analyze the initial and residual wastewater solution in terms of pH, Chemical Oxygen Demand (COD) and also turbidity.

1.4 Relevancy and feasibility of the project

The research is said to be pertinent and significant because, discharging the untreated textile wastewater to the surface water may bring serious destruction and bad consequences to the aquatic life and also to the people who lives in surrounding areas. In addition, there are number of cases that prove that improper expulsion of these wastewaters to the neighboring surface area may bring terrible impact to the neighboring communities. For example, in Mexico, fields and rivers which are located near the jeans factories are turning dark blue from untreated and unregulated dye effluent. Thus, the local residents and farmers report health problems and they do wonder either the food plantation in nearby fields is safe to eat or otherwise[8]. Meanwhile in China, one of the factories there dumps approximately 2,500 tonnes of wastewater containing dye a day into the river. As a consequence, part of the Yellow River turned to red in color[9].

Figure 1.2: Pollution in the Tehuacan Valley, Mexico due to runoff of dyes from a jean-manufacturing center[10]

Figure 1.3: Pollution in the Yellow River, China due to red dyes [11]

Besides, there are some researches that have been done previously, involving the textile wastewater treatment by implementing the coagulation and flocculation techniques. Thus, the research is done to further investigate on the suitability of the process in removing turbidity and reducing Chemical Oxygen Demand (COD) values from the wastewater solution containing of selected dyestuff. With a proper schedule, I believe that the research works can be accomplished within the time duration.

CHAPTER 2

LITERATURE REVIEWS

2.1 Legislations

In the early days whereby there are ample resources and negligible development, little attention has been given to the environmental issues. However, rapid economic development through the urbanization industrialization and the other land-use activities later on arises to water, air and land pollution, which remain as severe environmental problems in Malaysia. Thus, the government during 1974 has moved a step ahead by introducing a law and regulation, which is namely as Environmental Quality Act 1974 which functions in order to preserve and protect Malaysia's abundant natural resources. Its main purpose is to prevent, abate and control pollution, and further enhancing the quality of the environment in the country[12]. Pollution, as acknowledged in the legislation includes the direct or indirect alteration of quality of the environment or any part of it by means of a positive act or act of omission.

According to the Environmental Quality (Sewage and Industrial Effluent), the effluents from the wastewater treatment can be categorized into two, which are Standard A and standard B (refer to the Appendices). Standards A criteria applies only to the area located upstream of drinking water supply off-takes and meanwhile, Standard B applies for inland water[13]. In other words, Standard A will be applicable if the downstream of the river is used for the human activities, and vice versa for Standard B. Thus, this explains on why the Standard A appears much stricter compared to the other one.

2.2 Dyes

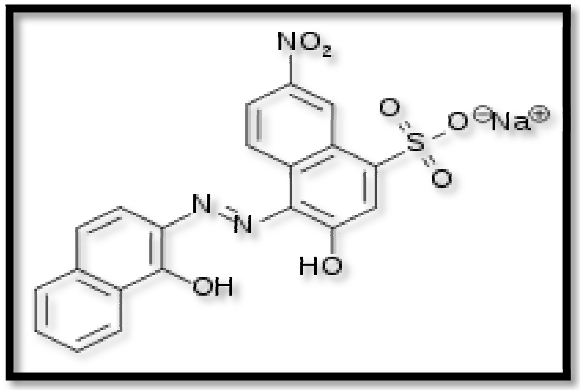

Figure 2.1: Structure of Eriochrome Black T[14]

Currently in the industry, there are more than 10,000 different types of dyes and pigments, which have been incorporated in the color index[15 - 16]. Based on these dyes, approximately 60 to 70 percent of them are from the azoic compound. In addition, azoic dyes have been used extensively in wide industries such as manufacturing of textiles, leathers, cosmetic, paper and so on.

In addition, azoic dyes are the one that contain nitrogen double bond in their structure. Normally, the color which may be produced by these dyes is determined through their azoic bond, association with chromophores and auxochromes[15]. Auxochromes basically are group of atoms which are attached to the chromophores and they can be categorized into two major classes[17]:

i. Acidic: - COOH, - OH, -SO_3H
ii. Basic: - NHR, -NR_2, - NH_2

Meanwhile, chromophores are the molecules which are responsible towards the color, whereby color may arises when the molecules absorb certain wavelength and reflects them[18]. Briefly, the most active bond in this kind of dyestuff is its azo bond itself, and the breakdown of these bonds may lead to the decolorization of dyes effluents. The azo bond can be weaken by means of reduced the electrons or broken down through the oxidation methods[15]. Besides, dyestuff can also be categorized in several ways, which are[19 - 20]:

i. Chemical structure (based on nature of their chromophores)
ii. Methods of dyeing
iii. Area and method of application
iv. Organic or inorganic
v. Natural or synthetic

2.3 Treatment technologies of dyes removal

Apart from the coagulation and flocculation method, there are various kinds of techniques which have been under research nowadays. In short, they can be classified in three groups, which namely as biological (e.g.: bacteria cell[21], fungi decolorization[22], etc), physical (e.g.: membrane[23], adsorption[19, 24, 25]) and chemical (e.g.: coagulation and flocculation[5, 6, 16, 26 – 29], ozone[26], electro kinetic coagulation[30],) treatments.

Throughout the research, coagulation and flocculation method is employed in order to eliminate the dyes from the wastewater. Despite of the production of sludge later on, the major advantages of this technology is that the decolorization process which is due to the dye removal, are not because of partial decomposition of dyes[27]. If so, it will have the probability to cause more toxic and harmful aromatic compound. In addition, there are also few researches which have been done under this field by using a variety of coagulants and flocculants such as aluminium salt, ferrous salt, chitosan, bentonite clay, and many others.

Besides, economic plays a vital factor in determining types of treatment that need to be applied. Even though few researchers claim that adsorption is well effective, but it is very expensive. And for membrane, it is not very practicable to be the first treatment due to the probability of membrane fouling, caused by particles or pollutants in the wastewater. According to few journal as well, biological treatment as the first treatment is not very feasible due to the characteristics of dyestuffs, which have low or poor biodegradability. Thus, this is the major reason on why coagulation and flocculation has been chosen as the first treatment apart from its cost efficiency and easy to operate.

Table 2.1: Comparison between various physical and chemical methods in dye removals[23]

Physical/chemical method	Advantages	Disadvantages
Fentons reagent	Effective decolourisation of both soluble and insoluble dyes	Sludge generation
Ozonation	Applied in gaseous state: no alteration of volume	Short half-life (20 min)
Photochemical	No sludge production	Formation of by-product
NaOCl	Initiates and accelerates azo-bond cleavage	Release of aromatic amine
Cucurbituril	Good sorption capacity for various dyes	High cost
Electrochemical destruction	Breakdown compounds are non-hazardous	High cost of electricity
Activated carbon	Good removal of wide variety of dyes	Very expensive
Peat	Good adsorbent due to cellular structure	Specific surface area for adsorption are lower than activated carbon
Wood chips	Good sorption capacity for acid dyes	Requires long retention times
Silica gel	Effective for basic dye removal	Side reactions prevent commercial application
Membrane filtration	Remove all dye types	Concentrated sludge production
Ion exchange	Regeneration: no adsorbent loss	Not effective for all dyes
Irradiation	Effective oxidation at lab scale	Requires a lot of dissolved O_2
Elektrokinetic coagulation	Economically feasible	High sludge production

2.4 Mechanism of coagulation and flocculation

Fundamentally, colloidal particles which have the size range between 10^{-6} to 10^{-3} mm, is the one that hold responsibility towards the high turbidity and color of the water. Apart from that, these particles will take approximately a year to settle a distance of only 1mm. Therefore due to the fact; these particles need to be eliminated from water and this is where coagulation and flocculation plays their major role. Colloidal system normally composed of two parts, which are dispersed phase and dispersion medium. And when there is discussion on the wastewater issue, our interest is normally focused on the solids which have been dispersed throughout the liquid. Colloid particles also can be categorized into two major classes, which are hydrophilic (has affinity towards water) and hydrophobic. In addition, the inorganic colloids are classified as hydrophobic and organic colloids are the way around.

Table 2.2: Types of colloidal system[31 - 32]

Dispersion Medium	Dispersion Phase	Common Name
Solid	Solid	Solid sol
	Liquid	Solid emulsion
	Gas	None
Liquid	Solid	Liquid sol
	Liquid	Liquid emulsion
	Gas	Foam
Gas	Solid	Gaseous sol
	Liquid	Gaseous emulsion
	Gas	Not applicable

Briefly, the coagulation and flocculation method is the techniques employed in order to eliminate the suspended solids or commonly, colloidal particles from the water solution. Even though the term of coagulation and flocculation is always used interchangeably, but yet, they are both referred to a two distinctive process. The detail process for both of them will be explained throughout this chapter afterwards. To sum up, the coagulation and flocculation occur in successive steps to overcome the charges or forces surrounding the particles so that, collision of particles are allowed. Later on, these steps may lead to the formation of sludge, which will be settled down and eliminated for the other treatment.

The first step of the process is destabilizing the particle's charges. Coagulants with the opposite charge with those of the suspended solids are added to the water and will start neutralizing these charges. Then, once the charges have been neutralized, the small suspended particles or the colloid particles are capable of sticking together to form microflocs, which are not visible to the naked eye. To promote better dispersion, collision and contact between these particles, high energy and rapid mixing is established during this stage. Standard mixing time for this period is up three minutes only.

After this stage has been completed, the flocculation stage is taking place. During this stage, the microfloc which are formed in the preceding stage are brought into contact with each other through the slow mixing. The major purpose of having gentle mixing rate is to avoid the shearing or breaking of the floc particles. If the breakage of floc is occurred, then it will be hard for them to be recombined again, and making the process becomes ineffective. The slow mixing then will allow the growth of the floc size, from microfloc to pin-floc and subsequently to macrofloc. Allowable contact time for this stage can be up to thirty minutes. Above and beyond, flocculants or coagulant aids may be added during this phase in order to help bridge, bind and strengthen the flocs, add weight and increase settling rate.

Following these steps, sedimentation process will take over. During this period, the produced floc will settle to the bottom part of the basin. Before water quality measurement is taken, the sludge and clear wastewater need to undergone the last process, which is filtration[31, 33].

Alike any other wastewater methods, this method is influenced by few parameters. The parameters can be one of the following:

i. pH of the solution
ii. Dosage of the coagulant and coagulant aids / flocculants
    Dosage basically associates in parallel manner with the concept of zeta potential, which refers to the colloidal particles and the surrounding particles. The greater zeta potential indicates greater stability and repulsion force. Thus, higher amounts of coagulants are sometimes necessary in order to overcome and weaken the forces[34].
iii. Operating temperature
iv. Mixing rate and detention time
v. Velocity

During the process, two other terms have been introduced, which namely as primary coagulants as well as coagulant aids (or sometimes known as flocculent). Following table will summarize the differences between these two materials.

Table 2.3: Comparison between coagulant and coagulant aids

	Primary coagulant	Coagulant aids
Requirements	• Is always required for the process.	• Sometimes, known as flocculants. • Is not necessarily required for the process.
Function (s)	• Neutralize the electrical charges of particles in the water. • Will causes the colloidal particles to clump together.	• Add density to the floc and toughness to the floc so that they will not break up. • Produce quick-forming and rapid-settling floc.

Normally in the real industry application, alum and iron salts are mostly employed due to the fact that, these kinds of coagulants can offer the lowest price per pound and is claimed to be quite effective in removing suspended solids[35]. Yet, consuming these materials may produce large amount of sludge and also may drop the pH throughout the process. Thus, the producible sludge from the experiment needs to be treated in an environmentally accepted manner to avoid any problems towards the surroundings.

Also, polyelectrolyte is becoming widely used nowadays, especially as coagulant aids together with the regular inorganic coagulants or as the primary coagulant too. As stated in Table 2.3, the use of polyelectrolyte as the coagulant aids will generate tougher and good settling floc. The polyelectrolyte can be either natural (e.g.: starch) or synthetic. The other material that may be used as the coagulant aids is bentonite clay. Literally, bentonite clay is used as the weighting agent in water that is high in color but low in turbidity and mineral content. This type of water usually would not form floc large enough to settle down. Thus, the bentonite clay will play its role in joining the small floc and make it heavier and subsequently, it can settle more quickly[31, 34].

In addition, consumption of polyelectrolyte through the process is claimed to be very much beneficial in terms of the following[35-36]:

i. Can reduce the production of sludge, from 50 up and up to 90 %.
ii. Sludge will contain less chemically bound water and may ease the disposal treatment afterwards.
iii. The requirements for alkalinity can be eliminated due to the property of polyelectrolyte which does not affect the pH.

Table 2.4: Common types of coagulants and coagulant aids/flocculants[34]

Chemical Name	Chemical Formula	Primary Coagulant	Coagulant Aid
Aluminum sulfate (Alum)	$Al_2(SO_4)_3 \cdot 14\,H_2O$	X	
Ferrous sulfate	$FeSO_4 \cdot 7\,H_2O$	X	
Ferric sulfate	$Fe_2(SO_4)_3 \cdot 9\,H_2O$	X	
Ferric chloride	$FeCl_3 \cdot 6\,H_2O$	X	
Cationic polymer	Various	X	X
Calcium hydroxide (Lime)	$Ca(OH)_2$	X*	X
Calcium oxide (Quicklime)	CaO	X*	X
Sodium aluminate	$Na_2Al_2O_4$	X*	X
Bentonite	Clay		X
Calcium carbonate	$CaCO_3$		X
Sodium silicate	Na_2SiO_3		X
Anionic polymer	Various		X
Nonionic polymer	Various		X

CHAPTER 3

METHODOLOGY / PROJECT WORKS

3.1 Summary of project works

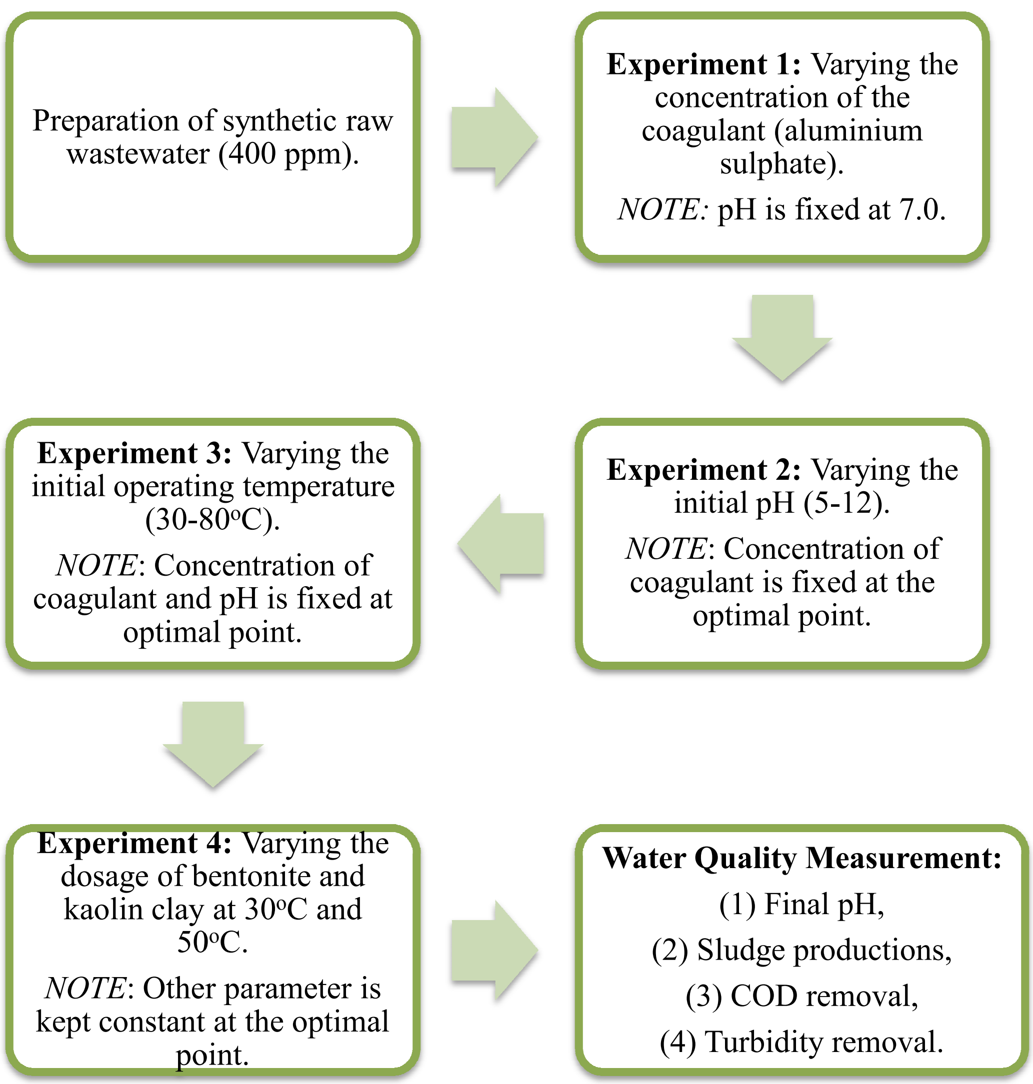

Figure 3.1: Project flow

3.2 Preparation of synthetic wastewater and materials

Before conducting the experiment, preparation of wastewater solution should be done first. According to literature, the standard textile effluent is much lower than 500ppm[37]. Thus, throughout the experimental works, 400ppm of dye concentration was chosen to be the initial concentration in accordance with the literature and also the previous journals. Associated with that, 1000 liter of distilled water was added to 0.4 gram Systerm StainPur™ Eriochrome Black T to dilute the solids. Apart from that, 0.1M of sodium hydroxide and calcium hydroxide were prepared also in order to control the pH during conducting the research works. Both of the sodium hydroxide and calcium hydroxide were supplied by Merck.

$$400\ ppm = \frac{400\ mg}{l} = \frac{0.4\ gram}{1000\ l} \qquad (Eqn.\ 3.1)$$

3.3 Experimental procedures

A standard jar test apparatus was implemented during the experimental works by varying few parameters, which namely as dosage of inorganic coagulants, aluminium sulphate (250 – 2000 ppm) and pH (5 – 12) and all of them were carried out at the ambient temperature (≈ 25°C). The FC6S-VELP *(*Scientifica*)* jar test principally was equipped with six paddle stirrer, together with six beaker apparatus. Apart from that, Julabo SW22 shaking water bath was also been implemented through the experimental works to determine the effect of various operating temperature (30 - 80°C) and dosage of coagulation aids, Aldrich PGV hydrophilic bentonite nanoclay and R&M Chemicals kaolin clay (in range of 250 – 1000 ppm) at 30 °C and 50 °C. Each beaker used for both jar test and water bath shaker apparatus was contained with 100 mL of synthetic wastewater prepared earlier.

Prior of pouring the wastewater into the beakers, the samples were mixed homogeneously to ensure a better dispersion of the dyestuffs. Then, the wastewater sample ought to be measured for turbidity and COD, representing its initial condition. During the experiments, the pH of the wastewater solution was controlled by the addition of calcium hydroxide or sodium hydroxide since the initial wastewater is acidic (≈ 4.90). Both of the process which were performed in batch mode was agitated at various mixing times and speeds which was rapid mixing at 200 rpm for 2 minutes after the preferred addition of inorganic coagulants (alum), subsequently followed by the slow mixing at 30 rpm for 30 minutes. Then, after the agitation was stopped, the suspension was allowed to settle down for another one hour. While conducting the experiments on the effect of the coagulation aids towards the process, the bentonite or kaolin clays were added into the system during the slow mixing phase.

3.4 Analytical analysis

Prior of measuring the final COD, turbidity and pH, the supernatants in each beaker were filtered by means of Advantec Glass fibre filter (47mm) and accomplished by the total suspended solids (TSS) apparatus. The main reason of applying the TSS apparatus and drying method (approximately at 105^{o}C for 24 hours) were to measure the produced sludge (without the moisture content) during the experimental works. Literally, the volume of produced sludge is part of major considerations when discussing about the coagulation and flocculation works since higher amount of the sludge may contributes to various difficulties to its disposal, handling and treatment at the later stage.[38]

During the experimental works, the pH of the wastewater was controlled and measured by using Eutech Std. Bench pH Meter Model 510. Besides, turbidity analysis was performed by applying HACH 2100P Portable Turbidimeter in which the wastewater was filled into the sample cell and put into the instrument cell compartment for the measurement analysis. Meanwhile, COD measurement was performed by means of HACH Digital Reactor Block (DRB) 200 and HACH DR5000 Spectrophotometer through colorimetric for high range COD (0-1500 mg/L) determination at the wavelength of 620 nm. This particular analysis which involves two different steps, digestion and COD determination commonly will estimate the total amount of the organic compounds in the water and its reagents consist of strong oxidants which oxidize the organic compounds to CO_2 and H_2O.

In addition, the percentage in reduction values for both of COD and turbidity is obtained according to the formula stated below. As information, C_o and C both indicate the initial and final values for COD and turbidity of the simulated wastewater, respectively.

$$Percentage\ removal\ (\%) = \frac{C - C_O}{C_O} \times 100\% \qquad (Eqn. 3.2)$$

CHAPTER 4

RESULTS AND DISCUSSIONS

4.1 Observations on color of the wastewater

When dealing with dye-containing wastewater, color commonly will be the first contaminant to be recognized of. As a matter of fact, the color contributed by the dyes, even in small quantities may provide a variety of harms such as reducing the penetration of the sunlight and therefore, may give a greater impact towards the living organism inside the river[39]. Thus, the coagulation and flocculation techniques is one of the preferred choice to overcome this visibility problems of the raw water.

(a) Raw wastewater (400 ppm)

(b) After the coagulation and flocculation process

Figure 4.1: Color of the wastewater solution

Despite of various coagulant dosage, pH, operating temperature and the coagulation aids dosage, the color of synthetic wastewater will always be reduced after the treatment, from the dark purple to the lighter color. The likewise observation on the color reduction through the coagulation and flocculation process can be seen from the previous journals as well. For instance, through research works done by H. Asilian *et al.*, approximately > 99% of color reduction may be achieved when applying the same method with the same coagulant (alum) and the tested wastewater was 400 ppm of acrylic water base color wastewater[40].

In addition, the color of the wastewater usually can be described as the indicator of the pollution level in the water. For instance, the dyeing process applied in the industry uses a broad number of synthetic dyes. This synthetic dyes on the other hands are manufactured through the addition of chemical agents to the natural dyes, which can be derived from organic and inorganic substances[23]. Associated with that, the relationship between the color intensity and the level of pollutants may be developed. By reducing the color of the wastewater, it can also reduce the number of contaminants inside the effluent stream. This statement can be proved in the next section whereby, we can see after the coagulation and flocculation process, the color of the solution becomes lighter and subsequently, the COD and turbidity values appears to be reduced as well.

The Eriochrome Black T (EBT) dyes – types of azoic dyes are applied throughout the experiment. The mechanism of decolorization of azoic dyes basically can be either oxidation by hydroxyl radicals or by electrons reduction[15]. However, from Figure 4.1, we can see that the final color of the wastewater solution are still not in the colorless form. This is might be due to the manufacturing process of the azoic dyes itself, which attempts to generate a coloring stability and therefore, the dyes are required to be resistant to biological attack, light, heat as well as oxidation.

Associated with that, degradation of these dye particles should be implemented. According to C. C. Z. Mutambanengwe *et al.*, azoic dyes which known for its water-soluble characteristics have a tremendously resistance towards the microbial and physical-chemical degradation[41]. Ordinarily, the decolorization for the typical azoic dyes involves two distinctive steps; (1) reduction of azoic groups to the bis-amine and (2) to the two separate amines[41]. Even so, there is an arguments proposed by Ganesh *et al.* states that the splitting of the azoic bonds (-N=N-) create a colorless aromatic amines[42].

Apart from that, J. Mo *et al.* recommend that color removal agent should be used together with the alum so that the colorant from the wastewater can be fully removed[16]. Hence, since the color itself is indirectly categorized as pollutant, the water treatment shall be implemented prior of releasing the wastewater into the nearby surface water.

4.2 Determination of optimum dosage of aluminium sulphate

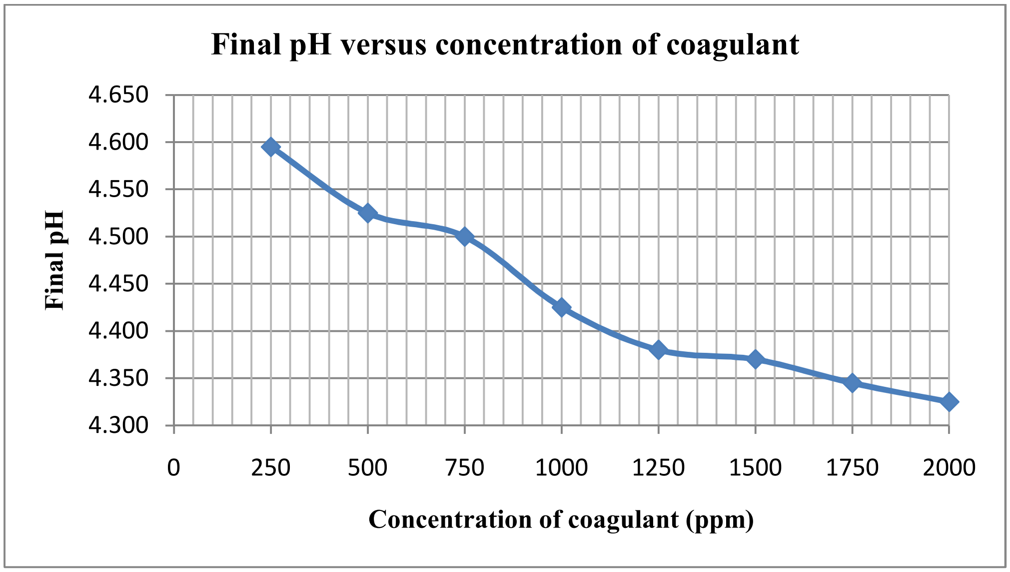

Figure 4.2: Final pH versus different concentration of coagulant

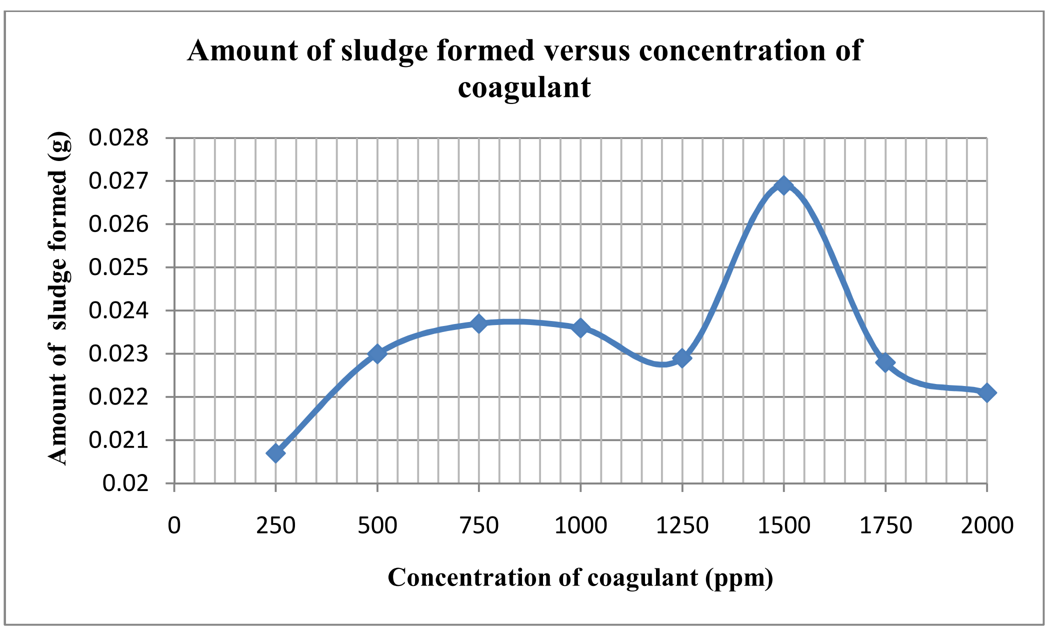

Figure 4.3: Sludge production versus different concentration of coagulant

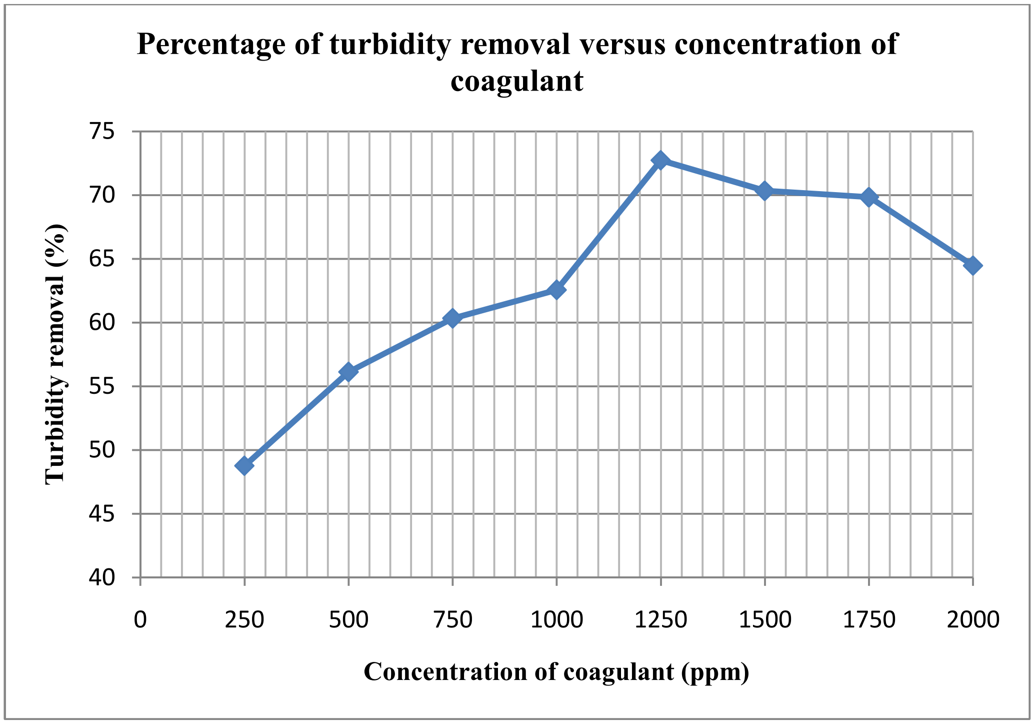

Figure 4.4: Turbidity removals versus different concentration of coagulant

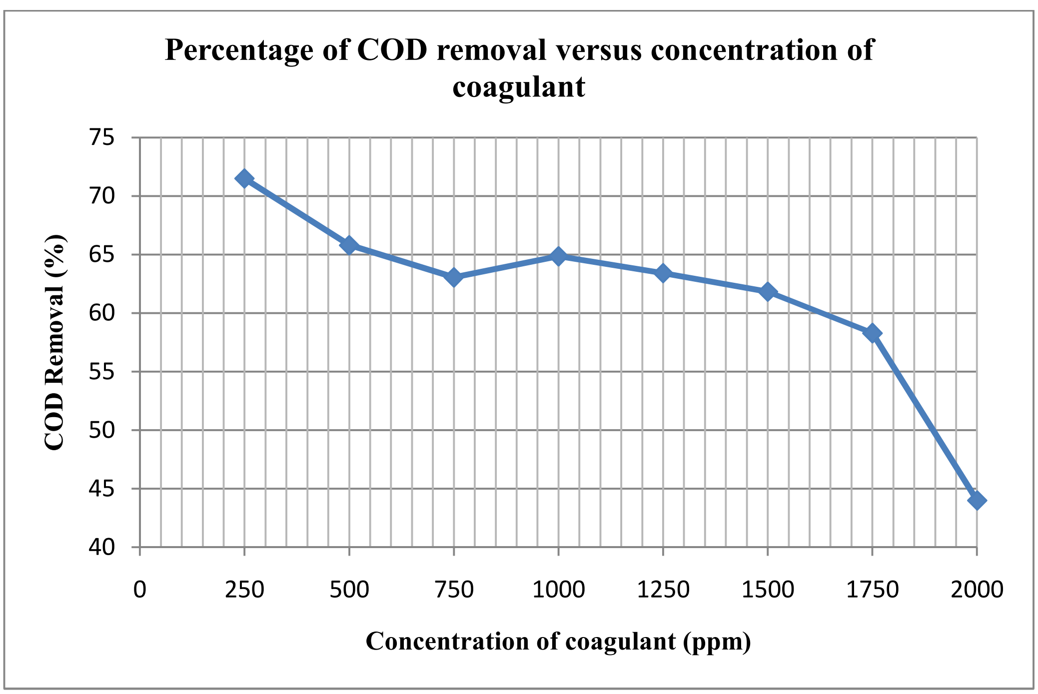

Figure 4.5: COD removal versus different concentration of coagulant

While dealing with the coagulation and flocculation experimental works, coagulant dosage is primarily one of the critical parameter in determining the effectiveness of the process. Thus, apart from the desire to achieve a better performance on the dye removal, the additional goals of achieving the optimal dosage point are to minimize the dosing cost as well as to reduce the number of sludge created during the process, due to the fact that these sludge might contributes to the handling and disposal problems afterward. In addition, prior of conducting the experiment, the pH of the dye-containing wastewater was controlled to be 7 by addition of $Ca(OH)_2$. In fact, this slaked lime will supply sufficient alkalinity to the wastewater so that the floc or precipitate can be created efficiently. Thus, the reaction that occurs during this process may be simplified as following:

$$Al_2(SO_4)_3 + 18H_2O + 3Ca(OH)_2 \rightarrow 2Al(OH)_3 + 3CaSO_4 + 18H_2O \quad (Eqn.\,4.1)$$

Through the above equation, it explains on the occurrence of precipitates, which is aluminium hydroxide, $2Al(OH)_3$ after the addition of coagulant into the wastewater. Associated with that, it is seen that volume of the sludge produced from this kind of treatment, which is without the addition of coagulation aid is barely around 0.02 to 0.03 gram. Besides, throughout the experiments, the wastewater quality is analyzed in accordance with Environmental Quality (Sewage and Industrial Effluent) Regulations 1979 as well as Interim National Water Quality Standards for Malaysia.

After the coagulation and flocculation treatment, final pH value seems to drop and it violates the legislation. This can be proved through the figures (refer to Figure 4.2, 4.6, 4.11, 4.15 and 4.16) and applied at all cases, despite of variance in coagulant dosage, pH, temperature or coagulant aids. Besides, this phenomenon can be explained by the following equation, which is the reaction between the alum and existing alkalinity inside the water itself.

$$Al_2(SO_4)_3.18H_2O + 3Ca(HCO_3)_2 \rightarrow 2Al(OH)_3 + 3CaSO_4 + 18H_2O + 6CO_2 \qquad (Eqn. 4.2)$$

From the equation above, it shows that the reaction may produce CO_2 gases along with the metal hydroxides. The gases then will react with water to produce carbonic acid, H_2CO_3. Therefore, it explains on the significant reduction of pH solution after the coagulation and flocculation process has been accomplished.

$$CO_2 + H_2O \rightarrow H_2CO_3 \qquad (Eqn. 4.3)$$

The results on turbidity and COD at different coagulant dosage were presented in Figure 4.4 and Figure 4.5, respectively. From figure 4.4, the curve of the turbidity reduction gives an N shape, which indicates that, the removal efficiency increase with increasing coagulant dosage at first (250 to 1250 ppm) and start to decline afterward. However, as the coagulant dosage keeps on increasing, the COD reduction is getting lower. Therefore, through some comparative study between the final COD and turbidity value with the current legislation, it is suggested that 250 ppm of alum is the optimum point. Above and beyond, even though the turbidity removal is not at the peak point at 250 ppm of coagulant, but still it does not violate the legislation.

M.H. Zoonozi. *et al* in their research affirm that the optimum point for alum in removing acid red 398 from 100 ppm dye-wastewater is at 120ppm[29]. On the other hand, Mo, J. *et al*., through the experimental works verifies that the optimum condition for the process to effectively occur is at 4000 ppm of coagulant dosage[16]. Thus, it can be suggested that dosage of the coagulant strongly depends on the initial concentration of dyeing wastewater or types of dyestuff applied. This argument is strongly supported by Klimiuk, E through his works, which clarifies that a higher initial concentration of dyeing wastewater, the requirement of alum coagulant dosage is increase as well[5].

From literature, the wastewater should be dosed at its optimal point so that the destabilization process of the colloidal particles can be effectively done and subsequently, flocculate into each other and settle down to the bottom part. But nevertheless, if the coagulant dosage goes beyond the optimum point, there is an possibility that the reverse process or restabilization phenomenon will take place. This argument is agreed by Mohd Ariffin, A.H. and colleague by affirm that if the coagulant dosage is beyond the optimum point, the poor performance of turbidity and COD removal occur due to the excess coagulant have been absorbed onto the colloidal surfaces[28]. As a result, the formation of inter-particle bridging is unable to be established due to the unavailability of site surface.

Even more, the colloidal particles which have been surrounded by the coagulant will be positively charged and may cause electrostatic repulsion between them. In addition, the higher dosage of coagulant might not necessarily contributes towards the better performance. This argument is strongly supported by J. Mo. *et al* as well as M. Malakootian that clarifies as the dosage increases, the electrical conductivity of the final solution will enhance too[16, 43].

4.3 Determination of optimum pH

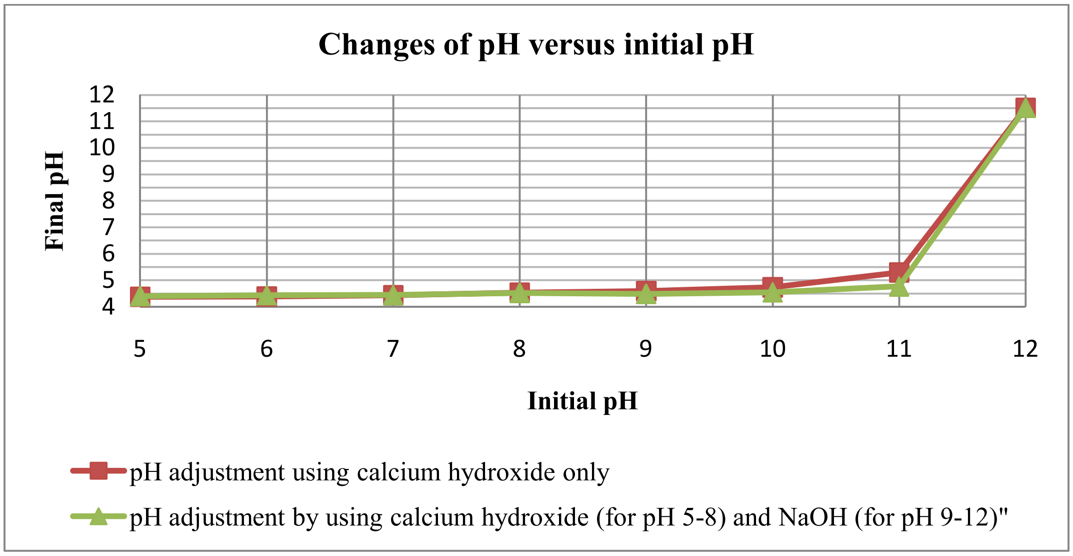

Figure 4.6: Final pH versus different initial pH

Table 4.1: Adjustments of pH by using calcium hydroxide and sodium hydroxide

pH	1st experiment	2nd experiment	
	Calcium hydroxide	**Calcium hydroxide**	**Sodium hydroxide**
5	< 0.1 mL	< 0.1 mL	-
6	0.2 mL	0.2 mL	-
7	0.6 mL	0.6 mL	-
8	0.8 mL	0.8 mL	-
9	1.2 mL	-	1 mL
10	1.6 mL	-	1.4 mL
11	3.0 mL	-	2.6 mL
12	16 mL	-	14.6 mL

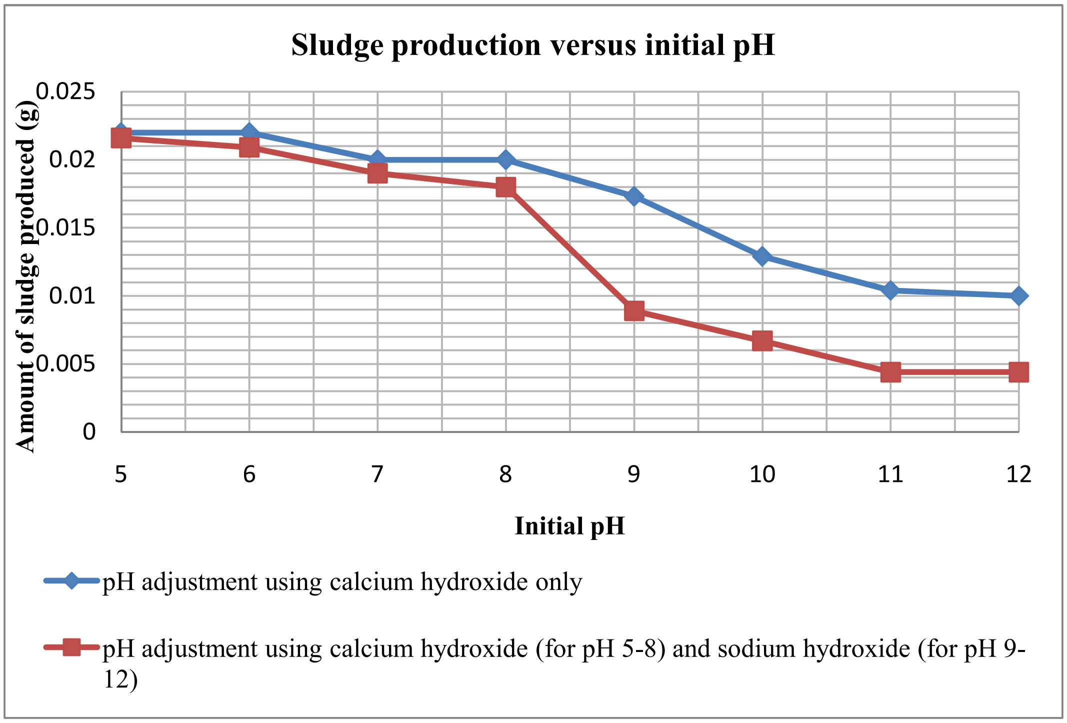

Figure 4.7: Sludge production versus different initial pH

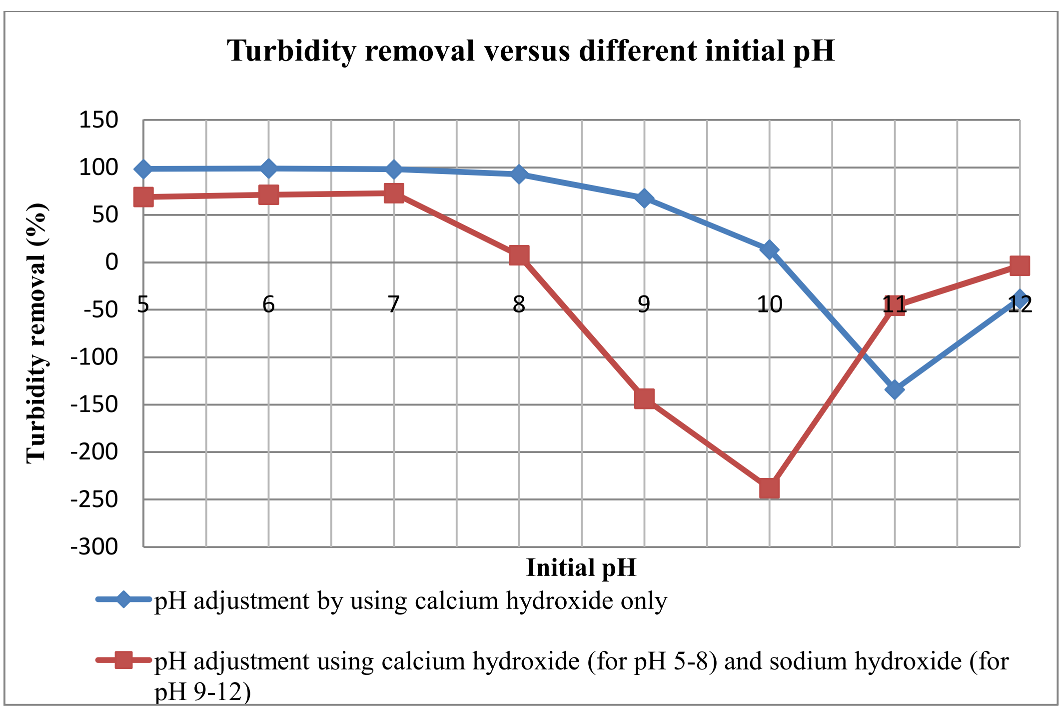

Figure 4.8: Turbidity removals versus different initial pH

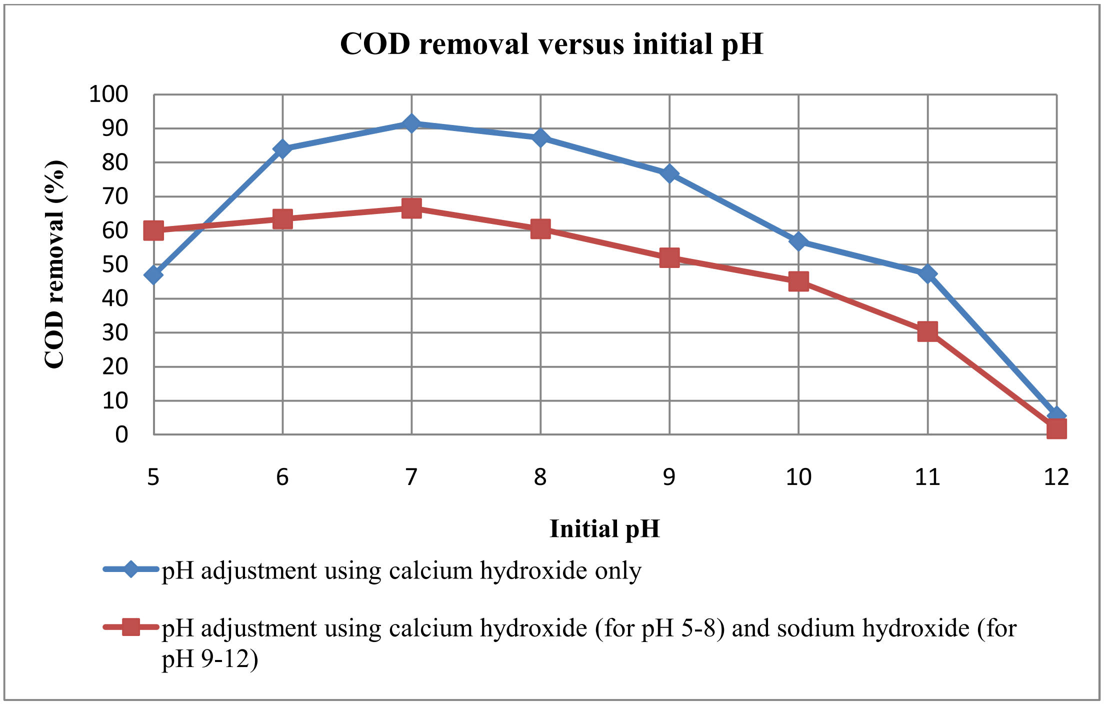

Figure 4.9: COD removal versus different initial pH

The initial pH of the dye-containing wastewater is approximately 4.90. Therefore, the pH of the wastewater samples are being controlled by the addition of caustic soda, NaOH liquid solution as well as slaked lime, $Ca(OH)_2$ liquid solution. In addition, prior of adjusting the pH, the hydrated lime should be maintained in suspension form and shall be agitated prior of being used during the experimental works. This is due to the characteristics of the solids, which attempts to settle down after awhile and forming two distinctive layers. Literally, the clear water (lime water) at the top part is just a dilution of the slaked lime and it may have lack of strength in altering the pH values. Therefore, if the slaked lime is not kept in suspension form and only part of lime water is used for pH alteration, it might increase its volume in adjusting the pH to the target value and consequently, might develop an inaccuracy or the coagulation process might not be much effective.

The basic idea on repeating the experiments with two different chemicals to alter the pH is mainly due to the initial hypothesis that 'the efficiency of lime attempts to decrease beyond 8.0'. According to an article, it states that the effectiveness of lime in adjusting pH is not so efficient above pH 8, which is probably because of some parts of the lime's surface area become less reactive. Associated with that, the volume or the addition of lime in controlling the pH might be slightly higher in order to overcome the problems. Therefore due to that reason, two sets of experiments have been carried out and the major concern is on the addition of both NaOH and $Ca(OH)_2$ after the pH 7.0. Apparently, from the Table 4.2, it can be seen that the volume of both chemicals while altering the pH value from 8.0 to 12.0 is nearly the same. Referring to the previous research, M. Malakootian *et al.*also use both NaOH and $Ca(OH)_2$ to serve the function as the softening agent and pH adjustment of the process[43].

Apart from that, Figure 4.9 and Figure 4.10 illustrates that the percentage reduction for both COD and turbidity appears to be incredibly higher when the pH of the wastewater solution is adjusted by using $Ca(OH)_2$ instead of NaOH. Yet, both of these trend lines provide the same pattern, which indicate that the removal percentage for both of the turbidity and COD parameters is the highest at pH 7.0. Thus, pH 7.0 can be portrayed as the optimum pH through the experimental works. Nevertheless, according to M. Malakootian *et al.*, the optimum pH for their research works (with same type of coagulant and effluent - 40 ppm of EBT wastewater) is turned out to be at value 11.15[43]. Besides, additional information which is attained through his research works is that the electrical conductivity of the final effluents increases with pH. Meanwhile, the deviation of the results for range of pH from 5 to 8 (in terms of turbidity and COD percentage removal) is proposed to be caused of the solids content of the $Ca(OH)_2$ solution itself. Even though from table 4.1, the volume of $Ca(OH)_2$ is nearly the same, but it does not guarantee the exact amount of solids of lime are added into the wastewater. Therefore, it is very crucial in performing the pH adjustment using lime since we should always maintain the solids in suspension. Likewise, maintaining the solids in suspension can become a drawback as well since if the solids settle down to the bottom part, it can affect the process performance.

Apart from being a softener agent and involving in pH adjustment, $Ca(OH)_2$ also may serve the function as the coagulant aids, which attempts to increase the density and toughness of produced floc so that any breakage of the floc will not be encountered during the mixing and settling process. Further, slaked lime also can increase the alkalinity of the solution and the increment in alkalinity contributes towards the enhancement of positive ions inside the water, which may react and attract the colloidal particles to generate floc. Thus, it explains on why applying slaked lime generates better efficiency compared to NaOH. In addition, this argument can be supported by Shamim Ara, who proposed that the combination of alum, lime and polyelectrolyte give higher percentage of turbidity reduction instead of using alum or lime alone[44].

In addition, variation of pH plays a major role in coagulation and flocculation process since it affects the charge on hydrolysis product and precipitation of metal hydroxide[29, 45]. This argument is approved by M. Ariffin *et al.*, who states that the pH will bring impact towards the surface charge of the coagulant and also stabilization of the suspension[28]. In accordance with E. Klimiuk *et al.*, they affirm that the stability of colloidal agglomeration depends on the forces that hold the particles in a suspension form. Even more, at the optimal pH, the coagulation and flocculation is much more efficient since the pH is adjusted to the iso-electric point, which enables these colloids to stick together[5]. In addition, the result for this experiment is analogous with the literature which verifies that the optimum pH for alum is in range of 4.5 to 8.0 due to the fact that alum hydroxide is insoluble within this range[33].

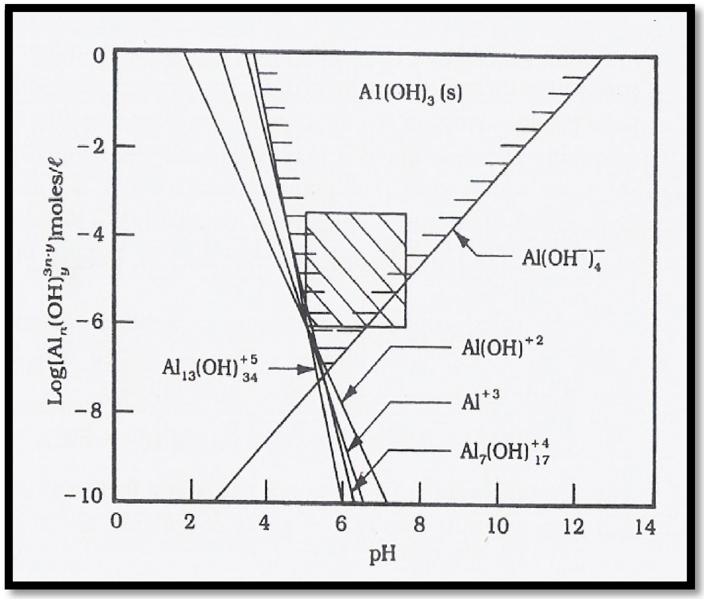

Figure 4.10: Solubility of aluminium hydroxide[14]

4.4 Determination of optimum temperature

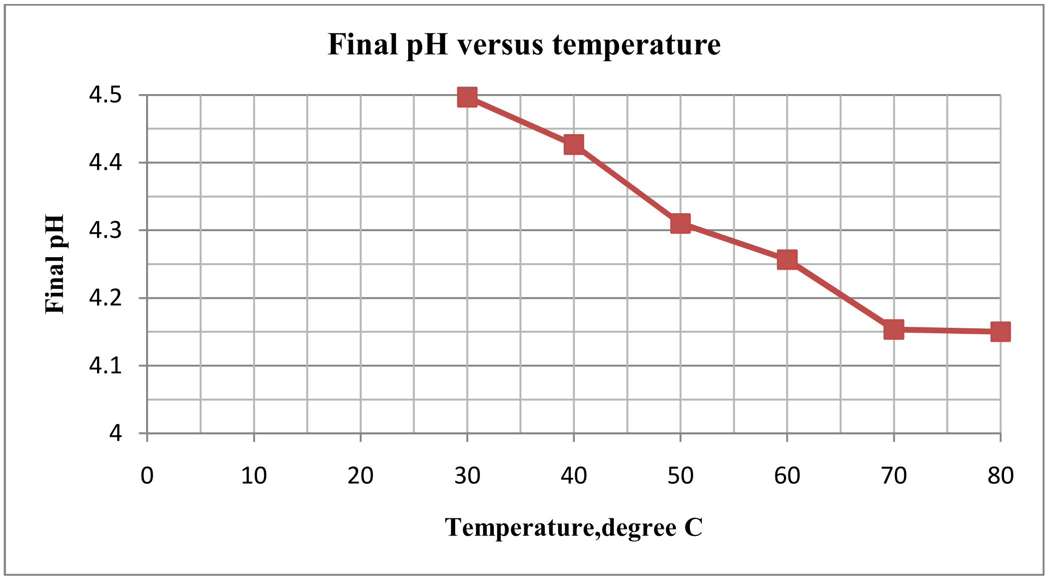

Figure 4.11: Final pH versus different temperature

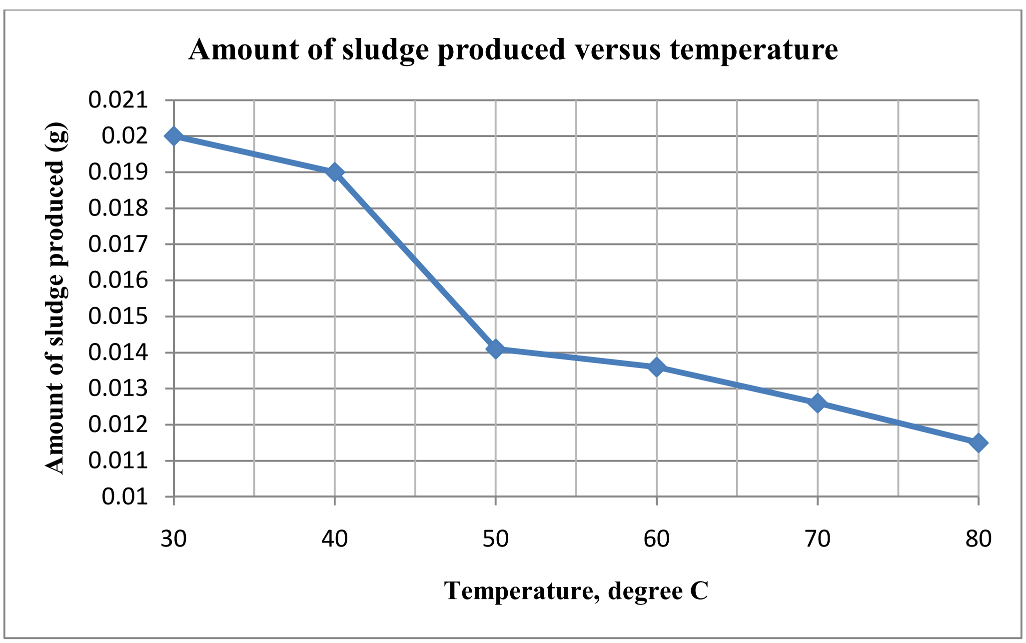

Figure 4.12: Sludge production versus different temperature

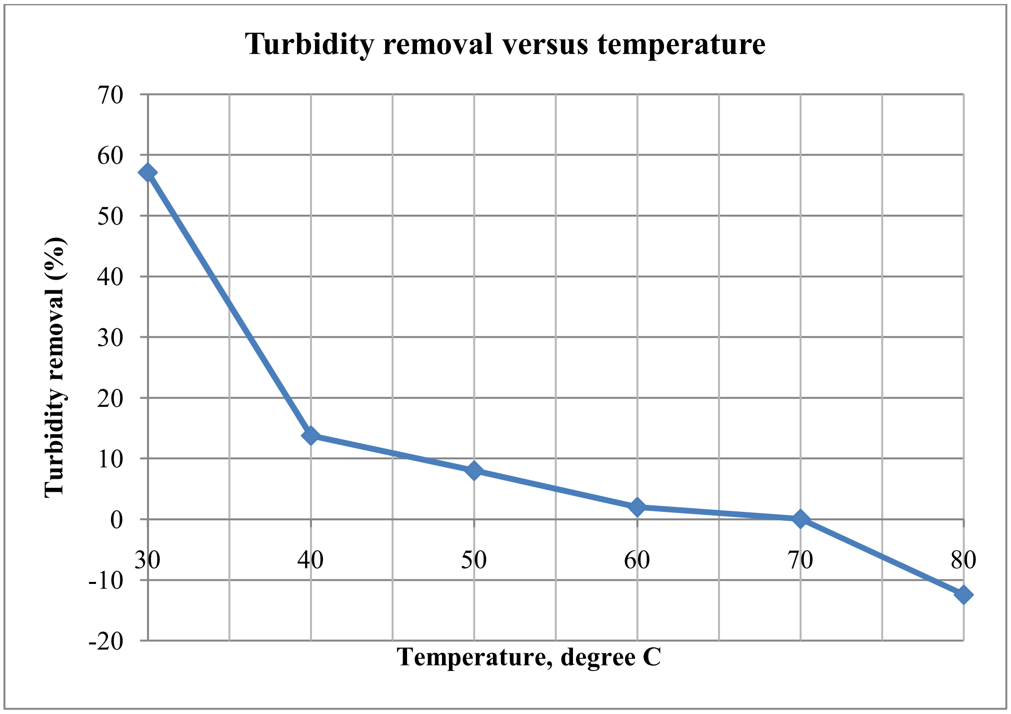

Figure 4.13: Turbidity removals versus different temperature

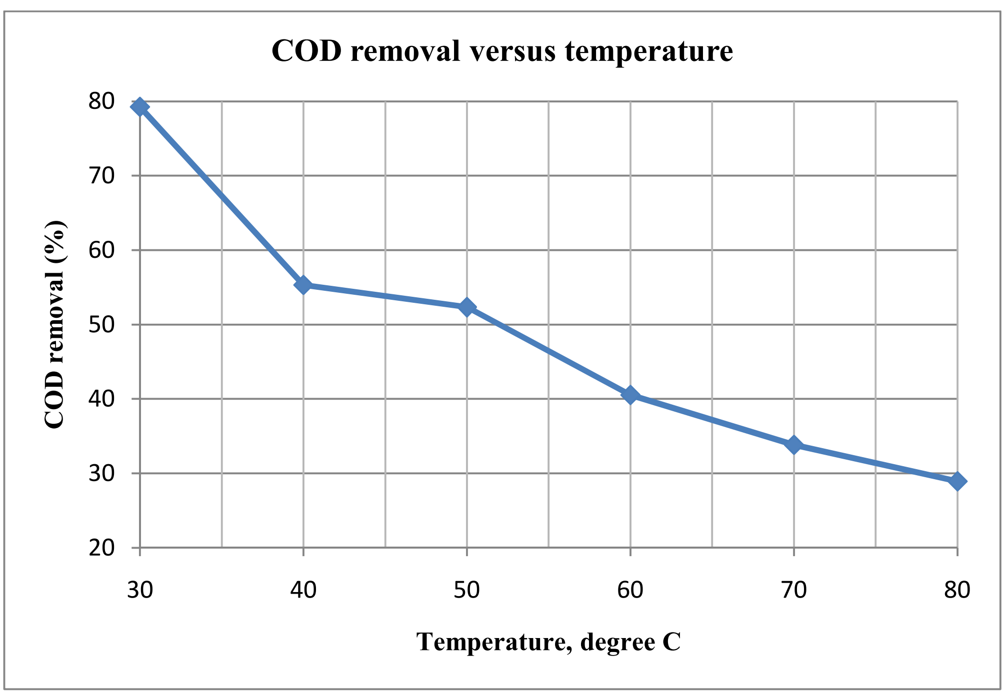

Figure 4.14: COD removal versus different temperature

Referring to Figure 4.13 and 4.14, it shows that by increasing the temperature from 30^oC to 80^oC, both of the turbidity and COD reduction is kept on decreasing. Also, from the observation done during running out the experiment, the color of the solution is turning on darker with the increment in temperature. Therefore, it can be assumed that at the higher temperature, there is a possibility that the formed precipitates re-dissolves into the solution. This argument can be strongly supported by looking through at Figure 4.12, which is the sludge produce at different temperature. At higher temperature, the sludge produced during the coagulation and flocculation process is kept on decreasing. Therefore, temperature 30^oC may represent the optimal point. Besides, there is a research that proposes that at higher temperature, the floc which are produced are somehow weaker and tends to re-dissolves into the solution once again.

However, the results of this experiment somehow contradict with the previous journal done by M. S. I. Mozumder and his colleague. Through their research works, the highest efficiency for dye removal (99%) was obtained at temperature 70^oC[46]. Principally, this research aligns with the concept of the higher the temperature, the reaction rate will be increase as well and subsequently, may results in more effectiveness of coagulation. When the operating temperature increases, it may enhanced the collision between the colloidal particles due to the enhancement of particles' kinetic energy, and afterward enable them to move faster.

4.5 Determination of optimum dosage of coagulant aids (flocculants)

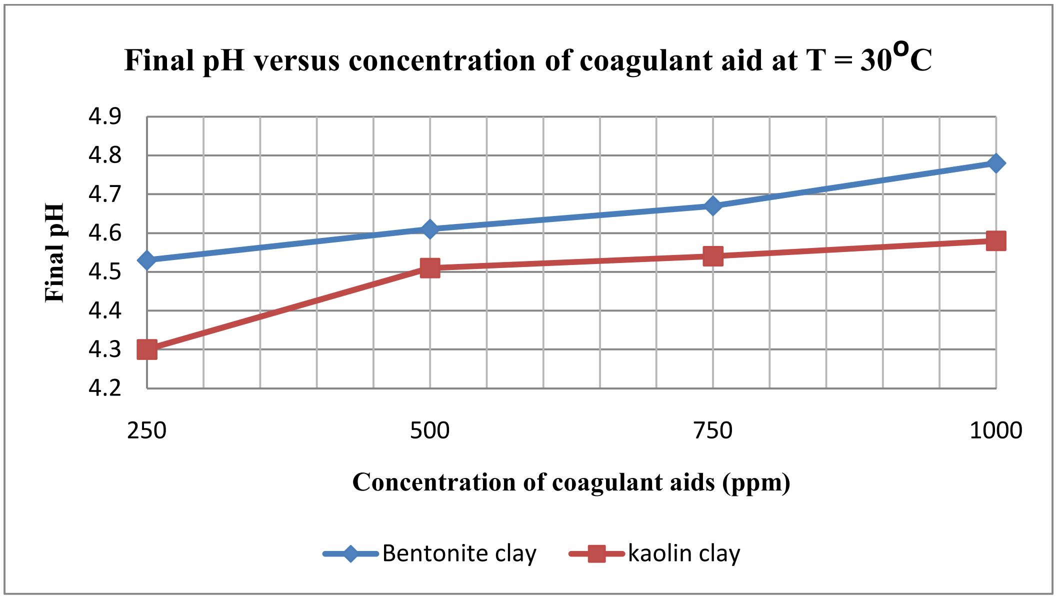

Figure 4.15: Final pH versus different concentration of coagulation aid at T = 30°C

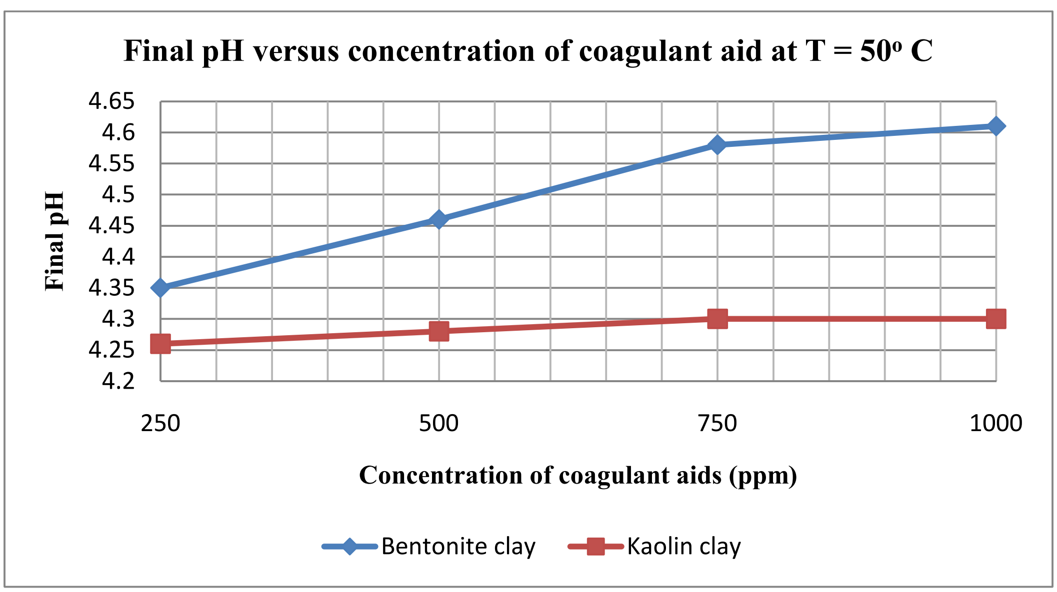

Figure 4.16: Final pH versus different concentration of coagulation aid at T = 50°C

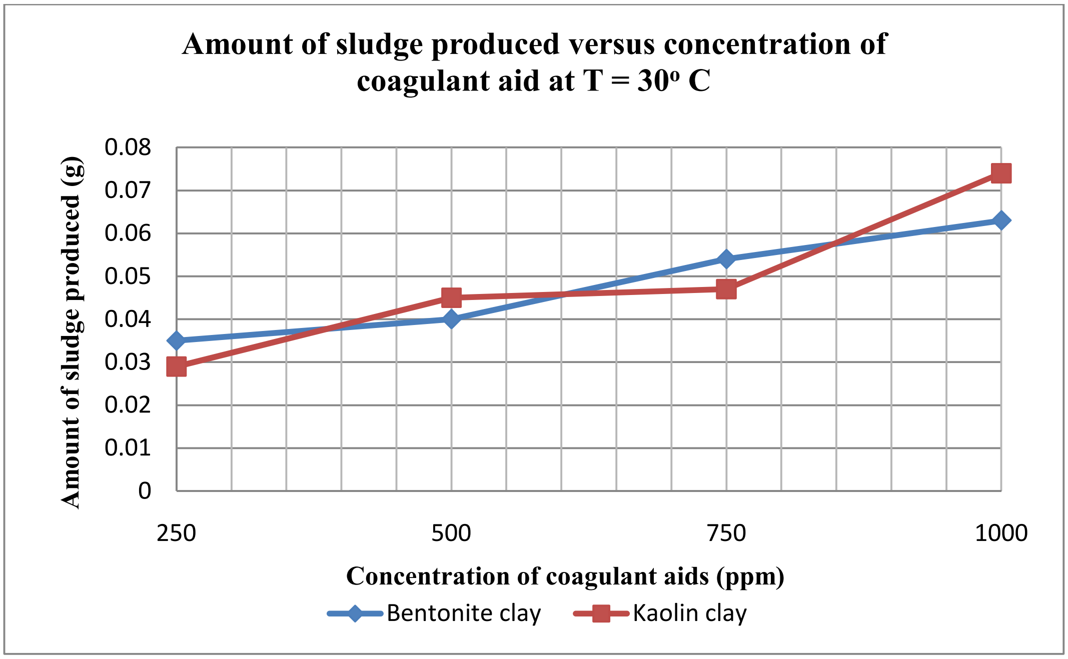

Figure 4.17: Sludge production versus different concentration of coagulation aid at T = 30°C

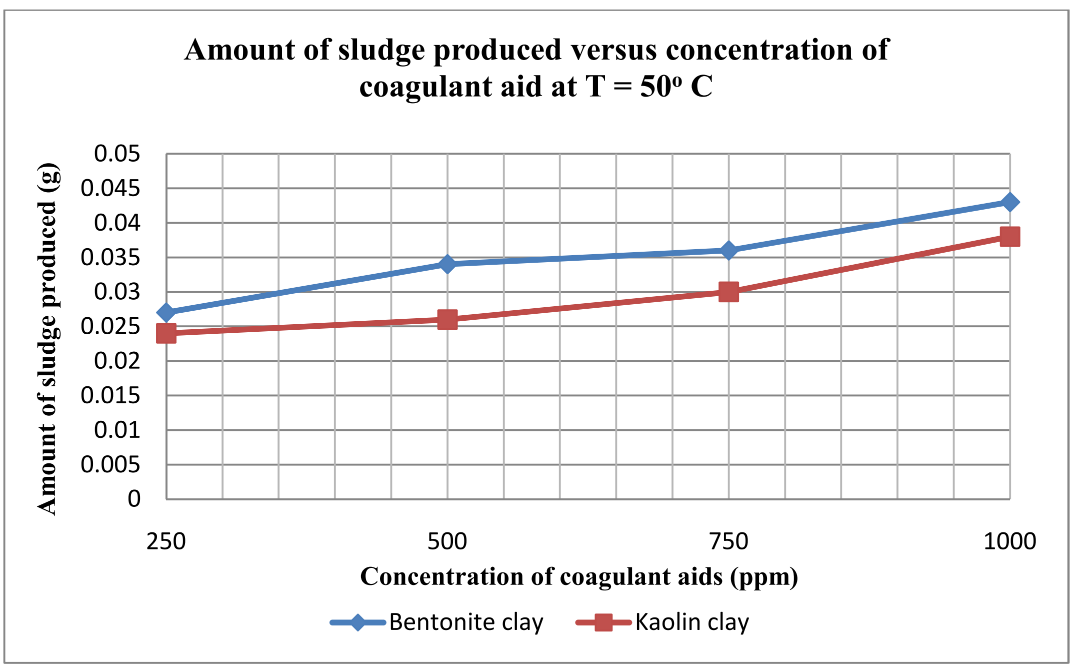

Figure 4.18: Sludge production versus different concentration of coagulation aid at T = 50°C

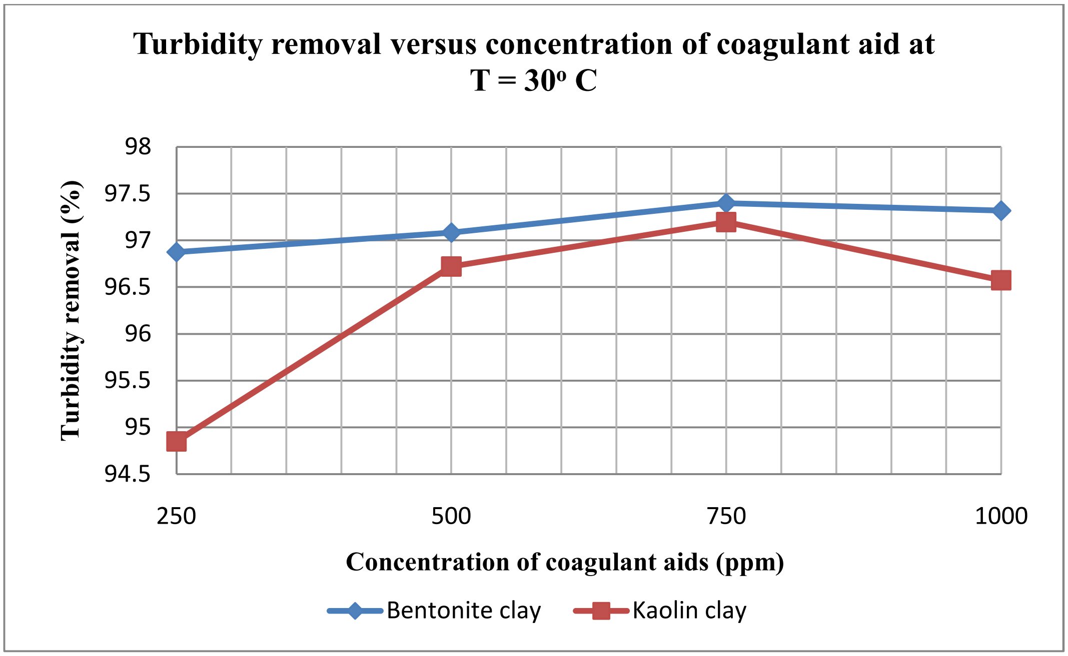

Figure 4.19: Turbidity removals versus different concentration of coagulation aid at T = 30^{o}C

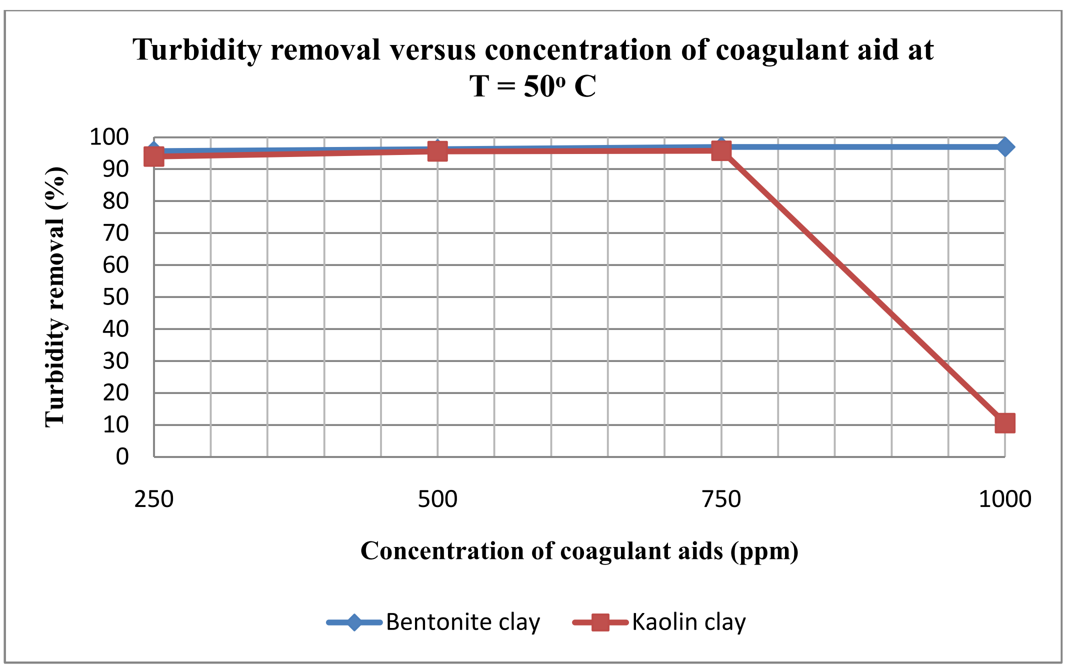

Figure 4.20: Turbidity removals versus different concentration of coagulation aid at T = 50^{o}C

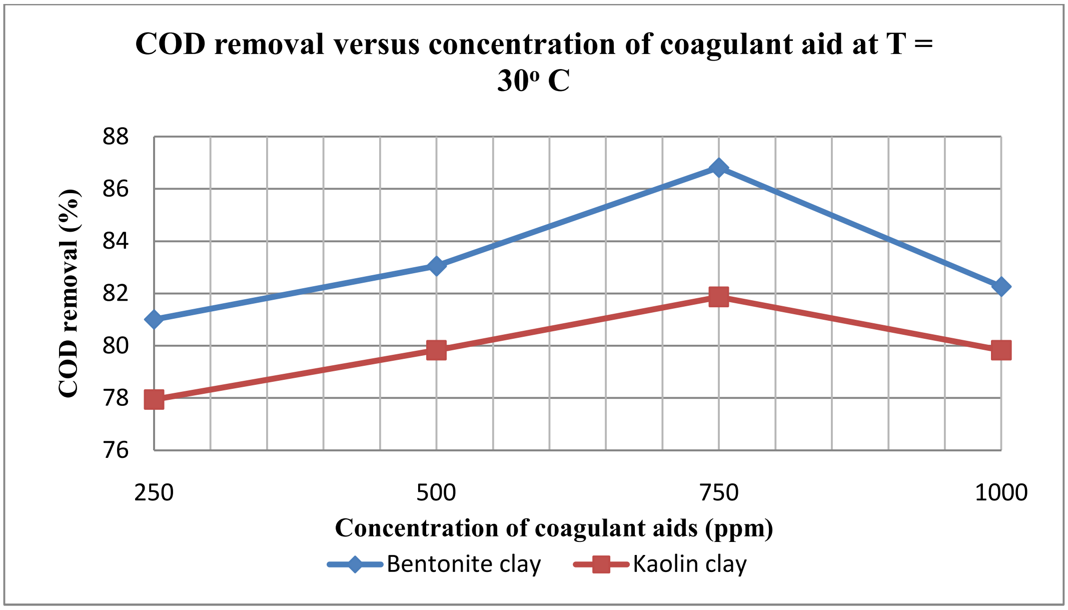

Figure 4.21: COD removal versus different concentration of coagulation aid at T = 30^{o}C

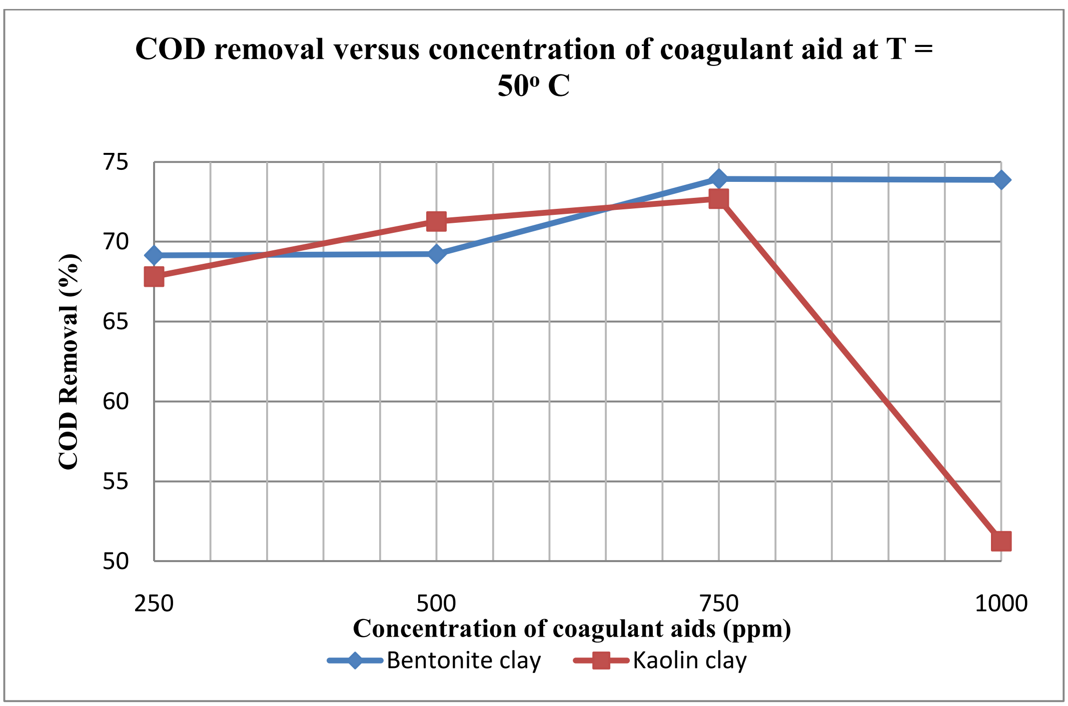

Figure 4.22: COD removal versus different concentration of coagulation aid at T = 50^{o}C

Coagulation aids such as polyelectrolyte, activated silica and clay are sometimes have been applied in the coagulation and flocculation process. As a matter of fact, these types of materials may enhance the toughness and settle-ability of the floc produced during the experimental works. Therefore, throughout this study, bentonite and kaolinite clay were elected, representing the natural coagulant aids. The experimental works basically were performed under two different temperature (30^{o}C and 50^{o}C) with different concentration of coagulant aids (250ppm to 1000ppm). While carrying out the experimental works, both of the coagulant aids will be added after the addition of alum into the dye-containing wastewater. This is essentially in accordance with the previous research done by B. Bina *et al*. who elucidate that coagulation aid should be added 1 minute after the addition of alum[47]. Otherwise, poor performance on the wastewater treatment may be encountered.

The results on the turbidity and COD percentage removal at temperature 30 ^{o}C are presented in Figure 4.20 and 4.22. Meanwhile, at the operating temperature = 50 ^{o}C, the results for turbidity and COD percentage removal are illustrated in Figure 4.21 and 4.23, respectively. After analyzing these figures, 750 ppm of kaolinite and bentonite clays may be established as the optimal concentration of coagulation aids. Besides, the performance of the percentage removal for both of COD and turbidity increase up to 750ppm and start to decline afterward. Besides, this experiment also can be claimed to be in accordance with the previous study (effect of temperature) despite of type of coagulant aids used in the research works. Above and beyond, from the figure itself, it can be seen that the removal percentage for turbidity and COD parameters are slightly higher at lower temperature, 30 ^{o}C instead of 50^{o}C. At 30 ^{o}C, the greatest turbidity and COD percentage for this case is obtained by applying bentonite instead of kaolinite clay, which is approximately 97.4% and 86.81 %, respectively. Looking back at Figure 4.14 and 4.15, the turbidity and COD greatest removal percentage is only at 57% and 79%. Therefore, it proves that the application of the coagulation aids may improve the performance, similar with other researches done[29, 48]. For instance, throughout the experimental works performed by M.H. Zoonozi, he is able to increase the dye removal from 18% (alum only) to 33% (alum-20ppm bentonite) [29].

From the result obtained through this study, the performance of coagulation - flocculation start to decline after 750ppm for both coagulant aids. As said by P.W. Wei *et al.*, over dosage or concentration may results in two dreadful impacts, which are contributing towards the increment of final sludge to be disposed afterward and also, may restrain the process. This is because if the volume of coagulant aids is too high, it may enhance the possibility of coating the colloidal particle with those coagulant aids. Thus, there might be insufficient of active surface site for the bridging process to occur[48].

Apart from that, the analysis demonstrates that bentonite clay is more efficient compared to the kaolinite clay in treating the synthetic dye wastewater. Fundamentally, the performance of both of these clays depends on the surface area as well as the exchange capacity. From literature, it states that bentonite has the capability to provide higher surface area compared to the other types of clay[49]. This is due to the characteristics of the bentonite itself, which have thinner sheet or layer of the atoms arrangement and is easier to be separated in the water. Therefore, it can be sum up that major the difference between the bentonite and the other type of clays is dependable on the lattice structure. In terms of cation exchange capacity occupied by both type of these clays, kaolin in general has less capacity for cation exchange, which is approximately around 2-10 meq/100g only compared to montmorillonite (major component in bentonite clays) which has the capability of 100 meq/100g[50].

In addition, the combination of clay-alum which produces significant results instead of applying alum alone also can be supported by F.B. Dilek and his colleague[51]. Throughout their research, they come out with proposals stated that the combination of alum and clay in treating the wastewater may be done through the charge neutralization and adsorption process. Meanwhile, the interaction which occurs between color (lignin) and alum is only due to the formation of floc only, and the color will be precipitated or adsorbed onto the floc. Thus, it can be concluded that the combination of alum and clay, either bentonite or kaolinite will give significant reduction since there will be two reaction takes place inside the solution, namely as charge neutralization and adsorption.

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

As acknowledged previously in the earlier chapter, textile industry is one of the major polluter in the worldwide since it consumed a high amount of fresh water as well as the chemicals, dyestuffs and auxiliaries. Accordingly, the problems that arise beyond the fact are such that, the textile manufacturing may produce effluent which deviate the local legislations[2]. Apart from that, there are varieties of treatment methods in treating the textile effluent. However, the coagulation and flocculation is preferred compared to the other methods because it is claimed to be economically feasible. In industry, cost plays a vital role and everyone in the industry strives to reduce the operational cost. Despite the fact that this kind of process will produce sludge and require following treatment, but if the coagulation and flocculation work efficiently at the optimal condition, it may ease the utilization or disposal of that sludge afterwards.

In summary, the coagulation and flocculation methods are only applicable at certain values of coagulant concentrations, pH of solution, temperature and also effect of coagulant aids.

i. The optimal concentration of coagulant which is aluminium sulphate is at 250 mg/l.
ii. The optimum pH solution is at pH 7. In addition, using calcium hydroxide (lime) instead of sodium hydroxide gives a similar reduction trend in terms of COD and

turbidity removal, but high efficiency is obtained by implementing calcium hydroxide.

iii. The optimum temperature is at 30°C, suggesting that the weaker floc will be achieved at higher temperature and dominant to re-dissolves into the wastewater liquid solution.

iv. For the coagulation aids, the optimum concentration is at 750ppm and bentonite clay will bring higher performance instead of kaolin clay due to surface area and ion exchange capability.

Overall, the combination of 250 mg/l aluminium sulphate in pH 7, temperature of 30°C as well as 750ppm of bentonite clay contributes to approximately 97.4% turbidity removal and 86.7% COD removal. In industry itself, jar test is very crucial to be done prior of carrying out the coagulation and flocculation process.

5.2 Recommendations

Through the research works, there are few recommendations which are proposed and can be applied for the future work undertakings. These recommendations are like the following:

i. While applying calcium hydroxide (lime) as the pH controller, the solution of the calcium hydroxide should be always thoroughly mixed to avoid any particle solids, $Ca(OH)_2$ from being settled down to the bottom part of the beaker.

ii. Further studies should be conducted regarding various methods of wastewater treatment of Eriochrome Black T dyes. Thus, the comparison in terms of COD and turbidity removal for each treatment may be investigated and developed.

iii. Otherwise, different types of coagulants can be tested for the same type of dyestuffs. Afterward, the contrast between the COD and turbidity performances can be assessed and evaluated.

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APPENDICES

THIRD SCHEDULE

ENVIRONMENTAL QUALITY ACT 1974

ENVIRONMENTAL QUALITY (SEWAGE AND INDUSTRIAL EFFLUENTS) REGULATIONS 1979

(REGULATIONS 8(1), 8(2), 8(3)

PARAMETER LIMITS OF EFFLUENTS OF STANDARDS A AND B

Parameter		Unit	Standard	
			A	B
(i)	Temperature	°C	40	40
(ii)	pH value	-	6.0 - 9.0	5.5 - 9.0
(iii)	BOD at 20°C	mg/ l	20	50
(iv)	COD	mg/ l	50	100
(v)	Suspended Solids	mg/ l	50	100
(vi)	Mercury	mg/ l	0.005	0.05
(vii)	Cadmium	mg/ l	0.01	0.02
(viii)	Chromium, Hexavalent	mg/ l	0.05	0.05
(ix)	Arsenic	mg/ l	0.05	0.10
(x)	Cyanide	mg/ l	0.05	0.10
(xi)	Lead	mg/ l	0.10	0.5
(xii)	Chromium Trivalent	mg/ l	0.20	1.0
(xiii)	Copper	mg/ l	0.20	1.0
(xiv)	Manganese	mg/ l	0.20	1.0
(xv)	Nickel	mg/ l	0.20	1.0
(xvi)	Tin	mg/ l	0.20	1.0
(xvii)	Zinc	mg/ l	2.0	2.0
(xviii)	Boron	mg/ l	1.0	4.0
(xix)	Iron (Fe)	mg/ l	1.0	5.0
(xx)	Phenol	mg/ l	0.001	1.0
(xxi)	Free Chlorine	mg/ l	1.0	2.0
(xxii)	Sulphide	mg/ l	0.50	0.50
(xxiii)	Oil and Grease	mg/ l	Not Detectable	10.0

INTERIM NATIONAL WATER QUALITY STANDARDS FOR MALAYSIA

		CLASSES					
PARAMETERS	UNIT	I	IIA	IIB	III	IV	V
Ammoniacal Nitrogen	mg/l	0.1	0.3	0.3	0.9	2.7	>2.7
BOD	mg/l	1	3	3	6	12	>12
COD	mg/l	10	25	25	50	100	>100
DO	mg/l	7	5 - 7	5 - 7	3 - 5	<3	<1
pH		6.5 - 8.5	6 - 9	6 - 9	5 - 9	5 - 9	-
Colour	TCU	15	150	150	-	-	-
Elec. Conductivity *	umhos/cm	1000	1000	-	-	6000	-
Floatables		N	N	N	-	-	-
Odour		N	N	N	-	-	-
Salinity (%)	%	0.5	1	-	-	2	-
Taste		N	N	N	-	-	-
Total Dissolved Solid	mg/l	500	1000	-	-	4000	-
Total Suspended Solid	mg/l	25	50	50	150	300	300
Temperature (C)	°C	-	Normal $+2^0C$		Normal $+2^0C$	-	-
Turbidity (NTU)	NTU	5	50	50	-	-	-
Faecal Coliform **	counts/100mL	10	100	400	5000 (20000) a	5000 (20000) a	-
Total Coliform	counts/100mL	100	5000	5000	50000	50000	>50000

NOTES:

N No visible floatable materials or debris, or No objectionable odor, or No objectionable taste.

* Related parameters, only one recommended for use

** Geometric mean

a maximum not to be exceeded

CLASS USES:

Class I	Conservation of natural water environment water supply 1 – practically no treatment necessary Fishery 1 – Very sensitive aquatic species
Class IIA	Water supply II – conventional treatment required Fishery II – Sensitive aquatic species
Class IIB	Recreational use with body contact
Class III	Water supply III – extensive treatment required Fishery III – common, of economic value, and torrent species livestock drinking
Class IV	Irrigation
Class V	None of the above